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PERFORMANCE OF METAL-CHROMIUM-ALUMINUM
(MCrAl), CERMET, AND MODIFIED ALUMINIDE
COATINGS ON IN-100 AND TYPE VIA ALLOYS
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ON IN-100 AND TYPE VIA ALLOYS AT 1093° C**

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16. Abstract <p>MCrAl, cermet, and modified aluminide types of coatings applied to IN-100 and NASA-TRW-VIA alloy specimens were cyclically oxidation tested in a high velocity (Mach 1) gas flame at 1093° C. Several coating compositions of each type were evaluated for oxidation resistance. The modified aluminide coating, Pt-Al, applied to alloy VIA proved to be the best, providing oxidation protection to approximately 750 hours based on weight change measurements. The second best, a CoCrAlY coating applied to VIA, provided protection to 450 hours. The third best was a cermet + aluminide coating on VIA with a protection time to 385 hours.</p>					
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HIGH-VELOCITY-OXIDATION PERFORMANCE OF METAL-CHROMIUM-ALUMINUM
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SUMMARY

Wedge bar test specimens of the nickel base superalloys IN-100 and NASA-TRW-VIA were commercially coated with metal-chromium-aluminum (MCrAl), cermet, and modified aluminide types of coatings. Various methods of application were used including plasma spraying, slurry dipping, physical vapor deposition, pack cementation, and combinations of these. The MCrAl compositions were FeCrAlY, NiCrAl, and CoCrAlY. The FeCrAlY and NiCrAl compositions were applied by plasma spraying; the CoCrAlY was physical vapor deposited. The cermets were applied by means of a slurry dip and vacuum sintered; then, in several cases, they were given aluminizing treatments by a standard pack method. Modified pack aluminide coatings were platinum-aluminum (Pt-Al), where the Pt was electroplated on the substrate then aluminized by a pack process, and, an aluminide coating containing embedded Al_2O_3 particles.

All specimens were cyclically oxidized at 1093⁰ C in a high-velocity gas flame (Mach 1). The Pt-Al coating on VIA proved to be the most oxidation resistant, surviving 750 hours before the onset of a weight decrease. The second best coating was the CoCrAlY composition applied to the VIA alloy. This was protective for 450 hours. The third best was an aluminized cermet coating on VIA with a protection time of 385 hours. All other coatings offered less oxidation protection.

It was also observed that, in general, all coatings provided a greater amount of protection for the VIA alloy. Most of the weight change-time data of the better coatings followed a curve with a generally parabolic shape.

INTRODUCTION

Advanced gas turbine engines require higher metal temperatures in order to in-

crease operating efficiency. For metal temperatures above 980⁰ C most nickel-base alloys used as blades and vanes in engines must be coated to resist oxidation-erosion and to extend their useful lifetime. The purpose of this study was to evaluate the high-gas-velocity (Mach 1) oxidation resistance of various coatings applied by commercial sources to two nickel base alloys: IN-100 and NASA-TRW-VIA. Wedge bar specimens (see fig. 1) of each alloy were sent to various coaters with the request that they apply, by any method, one or more coatings of their choice, either experimental or commercial in nature. The coatings that resulted are of three general categories: metal-chromium-aluminum (MCrAl), cermet, or modified aluminide. The MCrAl coatings were iron-chromium-aluminum-yttrium (FeCrAlY), nickel-chromium-aluminum (NiCrAl), and cobalt-chromium-aluminum-yttrium (CoCrAlY). (The NiCrAl being of proprietary compositions.) The cermet types were basically Ni-Cr alloys containing hard particles of oxides, borides, silicides, and nitrides. Some cermet coatings were also aluminized in a subsequent pack process. The modified aluminides consisted of Pt-Al, and an aluminide coating containing embedded Al₂O₃ particles. A variety of application methods such as plasma spraying, slurry dip and vacuum sintering, electroplating and aluminizing, and pack cementation processes were used.

The coated bars were exposed to cyclic oxidation in the high-gas-velocity (Mach 1) test facility at the Lewis Research Center. All tests were conducted at 1093⁰ C using cycles of 1 hour at temperature and 3 minutes cooling to room temperature. Performance of the coating-substrate systems were evaluated on the basis of weight change, thermal fatigue cracking, and various posttest microstructural examinations.

MATERIALS, APPARATUS, AND PROCEDURE

Specimens

The nominal composition of the substrate alloys are, for IN-100, nickel - 10-chromium - 15-cobalt - 5.6-aluminum - 3-molybdenum - 4.2-titanium - 1.0-vanadium and, for VIA, nickel - 6-chromium - 7-cobalt - 5-aluminum - 2-molybdenum - 1-titanium - 6-tungsten - 9-tantalum - 0.5-hafnium - 0.5-niobium - 0.4-rhenium. Both alloys were cast in the shape of wedge bars that were nominally 2.54 centimeters wide by 0.63 centimeter thick by 10.16 centimeters long with a 45⁰ leading-edge taper. The exact geometry of these bars is given in figure 1.

Bars of each alloy were sent to various commercial coaters who applied and processed their coatings. Table I lists the coating designations, coating application methods and processes, and compositions when available.

Of the MCrAl coatings, the FeCrAlY and NiCrAl (A) and NiCrAl (B) were applied by plasma spraying; the CoCrAlY coating was applied by a physical vapor deposition process. (See refs. 1 and 2 for further descriptions of the process.) The FeCrAlY coating was plasma sprayed as Fe-25Cr-13Al-0.8Y then oversprayed with aluminum powder followed by a 6-hour sinter in an argon atmosphere at 1100⁰ C. This resulted in a coating thickness of nominally 0.018 to 0.019 centimeter. The NiCrAl (A) and NiCrAl (B) coating compositions were proprietary. The NiCrAl (A) coating contained no added oxides; the NiCrAl (B) coating contained yttrium oxide (Y₂O₃) particles. These coatings were about 0.01 to 0.02 centimeter thick. The cermet coatings were applied by dipping the wedge bar into a slurry of the coating composition then firing in a vacuum for 1 hour at 1150⁰ C. The cermet (1) composition was a nickel-chromium-silicon alloy of unspecified composition containing titanium disilicide (TiSi₂) and titanium nitride (TiN) particles. The cermet (2) composition was a nickel-chromium alloy also of unspecified composition containing titanium diboride (TiB₂) particles. These coatings were about 0.008 to 0.013 centimeter thick. Coatings designated cermet 1A, 1B, and 2A were given an additional aluminizing pack treatment at Lewis after the commercial cermet applications. This processing is similar to that described in reference 3, where coatings of nickel-chromium were flame sprayed on steel substrates and then given aluminizing pack treatments. The pack used in the present study was composed of 2 weight percent aluminum, 2 weight percent sodium chloride, and 96 weight percent aluminum oxide (Al₂O₃). The pack time was 15 hours at 1090⁰ C in argon. Cermet 1A had an aluminum pickup of 15.3 milligrams per square centimeter, cermet 1B 13.0 milligrams per square centimeter, and cermet 2A 14.3 milligrams per square centimeter. These coatings ranged in thickness from 0.012 to 0.015 centimeter. The modified aluminide types of coatings were formed essentially by pack aluminizing. In the case of the Pt-Al coating a layer of platinum was first electroplated on the alloy substrate to a thickness of about 0.00076 centimeter. Then this was aluminized by an undisclosed pack process. This coating was nominally 0.008 centimeter thick. The embedded alumina particle aluminide (EAPA) coating was applied by a modified aluminizing pack process which also entrapped Al₂O₃ particles in the coating. (For more information on this process, see ref. 4.) The resulting EAPA coatings were nominally 0.003 to 0.005 centimeter thick. Coating thicknesses produced by the proprietary aluminizing pack process ranged from 0.011 to 0.013 centimeter.

High-Gas-Velocity Cyclic Oxidation

All test specimens were run in the same natural-gas-fueled burner rig operating with a gas velocity at the burner nozzle of Mach 1. A detailed description of the burner

conditions and photographs and drawings of the apparatus can be found in reference 1. In each test cycle the specimens were rotated in the air-rich natural gas combustion products for 1 hour at a metal temperature of 1093°C . The 45° tapered edges of the specimens were closest to the nozzle. At the end of this hour the specimens were lowered into a high-velocity (approximately Mach 1) cooling air blast for 3 minutes, which cooled the specimens to room temperature.

Specimen Evaluation

Weight change. - The test specimens were weighed to the nearest 0.1 milligram before the test and after every 20 1-hour cycles. The difference between the initial and subsequent weights of the specimen is the weight change used here. Up to 100 hours the results are based on the average of two specimens; above 100 hours the results are based on a single specimen.

Visual change. - All test specimens were also examined visually under a low-power ($\times 30$) magnification microscope at each 20-hour inspection for the appearance of thermal fatigue cracks and other surface changes. Macrophotographs were taken at appropriate intervals.

Other evaluations. - X-ray diffraction, microhardness, electron microprobe, and metallography were used to examine selected specimens both before and after testing. All procedures used were standard and widely employed in this type of specimen characterization; a discussion of them is presented in reference 1.

RESULTS AND DISCUSSION

Oxidation Test Data Presentation

High-gas-velocity, cyclic-oxidation weight change and thermal-fatigue-crack detection times for the Pt-Al coating on IN-100 and VIA alloys tested at 1093°C are presented in figure 2. The shape of these curves is typical of most of the oxidation weight change results of the coating-substrate systems studied. An examination of the curves revealed a general and distinctive shape with certain distinguishable features. The first is that at some time a maximum in the weight gain occurs and after this time the specimen always shows a weight decrease. Second, at some later time the weight change curve crosses the zero weight change axis. Third, the slope of the curve between these times varied depending on the coating-substrate system. Last, the maximum weight gained and time to the first detectable thermal fatigue crack varied with the coating-

substrate system. As a result of these general observations a schematic oxidation weight change curve was constructed and several parameters defined. This schematic curve is presented in figure 3. Parameter t_1 is the time to reach the maximum weight gain, t_2 is the time of crossing the zero axis, and the slope is defined as the slope of the straight line connecting the weight change values at the t_1 and t_2 points. This slope is given in units of milligrams per hour, and it has a negative sign since it always represents a weight loss. Values of t_1 , t_2 , slope, maximum weight gain, and time at which the first thermal fatigue crack was detected were taken from the experimental observations and the weight change curves of each coating-substrate system. These values along with the metallographically measured nominal coating thicknesses before oxidation are tabulated in table II. In this table the coating-substrate systems are arranged in increasing order of parameter t_1 .

It will be noted in table II that in several cases the values of t_1 and t_2 were not determined. These are cases where a weight loss was recorded at the first inspection time of 20 hours. It is probable that at least several systems may have exhibited a weight gain in this initial 20-hour period and t_1 and t_2 were very small. The slope value reported in table II for these systems was calculated from the initial weight loss of the specimens. These slope values are used as a comparative measure of oxidation.

Comparison of Coating-Substrate Systems

Since the primary objective of this study was to identify the most oxidation resistant coating-substrate systems, some means of judging the performance of various systems was needed. Since t_1 is the time at which the specimen begins to show a weight decrease (or shows a maximum weight gain), this is assumed to be the beginning of the loss of coating protection or the onset of degradation of the coating-substrate system in this series of tests. This weight decrease is caused by the fact that spallation or vaporization is now great enough to cause a net weight loss of the specimen. The beginning of degradation of the coating has actually been metallographically observed in several specimens examined near t_1 . One such example will be presented in later discussion. Assuming that t_1 is at or near the start of coating degradation, those coating-substrate systems exhibiting the larger values of t_1 are judged to be the better systems. Since the coating-substrate systems in table II are arranged in the order of increasing t_1 , then the better systems are at the bottom of this table. Based on this argument the best systems are the Pt-Al/VIA, CoCrAlY/VIA, and cermet 2A/VIA systems. The prevention of thermal fatigue cracking will be a secondary criteria. A long time to thermal fatigue cracking is considered here to be a desirable but auxiliary factor. It could not be used as a primary factor because the time to cracking was not determined in every

case because of the termination of testing caused either by large weight losses or for other reasons.

It is further interesting to note that the three best coating-substrate systems represent coatings of different types and application methods. The Pt-Al coating is a modified aluminide type applied by a dual process of electroplating followed by pack aluminizing. The CoCrAlY coating is an MCrAl type applied by physical vapor deposition. And the cermet 2A is a slurry applied, vacuum sinter cermet coating followed by a pack aluminizing. The only things in common are that they are all on VIA alloy and all contain aluminum.

A plot of the parameters t_1 , t_2 , and time to first detectable thermal fatigue crack grouped on the basis of the substrate alloy is presented in bar graph form in figure 4. From an examination of this presentation, it is easy to determine that most coatings performed better when applied to alloy VIA than IN-100 with respect to both t_1 and fatigue time. This difference in coating performance is believed to be due in part to the relative oxidation resistance of the bare substrate alloys. The data in table II for the bare IN-100 and VIA alloys show that alloy VIA has a smaller slope value than IN-100. The difference in performance is probably also related to differences in chemical or physical properties of the substrates which alter these interactions with the coatings. For example, the VIA alloy may provide a diffusion barrier to slow the diffusional destruction of the coating. In any event the same coating applied to different alloys does not perform in the same way.

These conclusions give no consideration to coating thickness variations between systems or application methods. It can be argued that, for other coating thicknesses or methods of application, the results would have been reordered. For example, the plasma sprayed FeCrAlY, NiCrAl (A), NiCrAl (B), and unmodified cermet coatings on both substrates peeled off rapidly in the highly erosive, high-velocity atmosphere used here because of the porosity, cracks, and low adherence of these coatings. Based on these observations it is probable that the use of other application methods or even different plasma spray or cermet coating parameters would change the ranking of these coating-substrate systems. Also, the use of other criteria for judging or ordering the systems could produce different conclusions as to the best systems. However, the criterion used here is reinforced by the metallographic observation of actual cases of coating breakup at or near t_1 .

General Results

As an extension of the specific considerations of the parameters in table II, an examination of this table for any general interrelations of these parameters was made.

This was done by cross-plotting the parameters. Two relations were found, and they are presented in figures 5 and 6. Figure 5 is a plot of parameters t_1 against t_2 . The slope of the line in this figure is approximately two. Therefore, a general or approximation relation of $t_2 \approx 2t_1$ exists between these two parameters. Pursuing this observation a little further on a geometric basis leads to the conclusion that most of the weight change results can be represented by a parabola opening downward with the axis of symmetry at t_1 . For example, using the values of t_1 , t_2 , and the maximum weight gain given in table II for the EAPA/IN-100 system, the following polynomial was determined:

$$y = -8.8 \times 10^{-4} x^2 + 0.238x$$

where y is the weight change of the specimen and x is the time in hours. These observations may be useful, in more sophisticated form, as a part of a predictive method that could be used to estimate life remaining in the coating-substrate system from current or short time data.

Figure 6 is a plot of the weight change slope parameter against the time to thermal fatigue crack detection. Even with the considerable degree of scatter of the points, it appears that a relation exists between these parameters: a decrease in slope leading to an increase in time to thermal fatigue. Since the slope parameter, in physical terms, is weight change per unit time (mg/hr) and since its value is negative, or a weight loss, the slope parameter may be a wearout factor of the coating-substrate system due to diffusional-spall destruction of the coating layer. Therefore, this would suggest that the more slowly wearout occurs, the greater the time to the thermal fatigue crack. This discussion would appear to preclude thermal fatigue cracking before t_1 , but crack times less than t_1 were observed for three systems, the Pt-Al/VIA, EAPA IN-100, and NiCrAl (A)/VIA. All the other systems have thermal fatigue crack times greater than t_1 and some are equal to or greater than t_2 . No explanation for the three exceptions can be given, but coating brittleness may be a factor for future consideration. Also, at the other extreme of thermal fatigue cracking, that is, cracking occurring at times far in excess of t_2 , a clear explanation cannot be given. This would imply thermal fatigue protection is provided long after total coating destruction.

Detailed Results for the Pt-Al/VIA System

Since the Pt-Al/VIA system proved to be the best system tested, a further analysis of the pretest and posttest properties appears warranted. Metallographic, microhardness, X-ray diffraction, and electron microprobe examination results of a pretest

specimen and a specimen tested for 1020 hours in the high-gas-velocity test rig will be given. A detailed discussion of the second best system, namely, the CoCrAlY/VIA system, can be found in reference 1. However, a photomicrograph of the as-coated and oxidized structure of this system will be presented later for convenience. A photomicrograph of the third best system, the aluminized cermet, in the as prepared and oxidized condition will also be presented later.

The microstructure of the pretested Pt-Al/VIA specimen is presented in figure 7(a). It is a multilayered structure similar to many coatings. X-ray diffraction of the surface of the pretested specimen revealed two phases to be present: the major phase was platinum aluminide (PtAl_2) and the minor phase was of a β -nickel aluminide ($\beta\text{-NiAl}$) structure. The first layer has a microhardness of 500 Knoop at a 50-gram load. The second layer has a hardness of 520 Knoop, and the diffusion zone adjacent to the substrate is 905 Knoop, both at a 50-gram load. The integrated scan (160- μm scan width) electron microprobe traces are presented in figure 8. Figure 8(a), for the pretested specimen, reveals a surface layer rich in platinum-aluminum and some nickel. On progressing farther into the coating, the platinum, aluminum, and nickel increase, suggesting a layer of PtAl_2 and NiAl extending up to 20 to 30 micrometers into the coating. Further into the coating the platinum decreases very rapidly and is not detectable at about 40 micrometers. At the same time, the nickel and aluminum are reaching a peak. This suggests a narrow layer of NiAl centered near 30 to 40 micrometers. The chromium content increases rapidly at depths from 40 to 50 micrometers, and the nickel and aluminum levels are decreasing to the concentration levels of the substrate.

The microstructure of the coating after 1020 hours of high-gas-velocity testing is presented in figure 7(b). The coating is in some intermediate stage of degradation, it is no longer a continuous coating layer but has broken up into islands dispersed in a matrix. The islands have a Knoop hardness of 420, and the NiCrAl matrix has a hardness of 600, both at a 50-gram load. After 1020 hours the specimen is between the weight change parameters of t_1 (750 hr) and t_2 (1400 hr) (see table II): This microstructure agrees with the premise given previously that the parameter t_1 is the time at which coating degradation begins. X-ray diffraction of the surface of this specimen revealed Al_2O_3 as the major phase with some Ni (solid solution). The integrated scan microprobe results are presented in figure 8(b). A thin surface layer rich in aluminum and oxygen, which is due to the Al_2O_3 scale, was detected. The platinum, nickel, and chromium in the surface layer are very small, but at about 10 micrometers these elements increase rapidly in abundance, while the aluminum decreases. The abundance of nickel, chromium, aluminum, and platinum remain constant out to approximately 50 to 60 micrometers where nickel, chromium, and aluminum become equal to that in the substrate. The platinum decreases beyond 60 micrometers but does not become zero

even at 120 micrometers. These results indicate that the islands are probably PtAl_x with a surrounding alloy of NiCrAl alloy.

Figure 9 presents macrophotographs of the side surface and trailing edge surface of the Pt-Al/VIA specimen after 424, 840, and 1020 hours of high-gas-velocity cyclic testing. At 424 hours no leading- or trailing-edge cracks were detectable to the unaided eye; however, at a magnification of $\times 30$ a small trailing-edge crack was detected. By 840 hours of testing both a leading- and trailing-edge crack were clearly visible. The trailing-edge crack appeared to be more of a tear in the coating than a crack that extends into the metal substrate. By 1020 hours both the leading-edge crack and the trailing-edge tear had increased in extent and some pitting of the trailing edge was now also present.

The microstructure of the second best coating system, namely, the CoCrAlY/VIA, in the pretested state, is presented in figure 10(a). It is a rather thick coating composed of β -CoAl and Co (solid solution). Figure 10(b) shows the structure after 600 hours of cyclic oxidation. It is apparent that the coating has undergone degradation. Some localized regions exhibited oxidation attack deep into the coating. Aluminum oxide was generally observed as the major surface oxide.

The microstructure of the third best coating system, the aluminized cermet (Cermet 2A) in the pretest condition is presented in figure 11(a). A layer of β -NiAl is present at the surface with a complex structure beneath. The coating structure after 622 hours of cyclic oxidation is presented in figure 11(b). It is evident that considerable degradation of the coating has occurred, but islands of the β -NiAl phase are yet present.

SUMMARY OF RESULTS

This work was initiated to evaluate the oxidation performance of some current commercial and experimental coatings on two nickel-base substrate alloys, namely, IN-100 and NASA-TRW-VIA. The objective was to identify the most oxidation resistant coating-substrate system procured. Three general types of coatings were applied: the metal-chromium-aluminum, cermet, and modified aluminide types. Methods of application used were plasma spraying for the FeCrAlY and NiCrAl coatings, physical vapor deposition for the CoCrAlY, slurry-dip followed by vacuum sintering for the cermet coatings, and modified pack aluminizing processes for the EAPA coating. Testing was done in a high-gas-velocity (Mach 1) burner rig using a natural-gas fueled burner. Tests up to 1020 hours at 1093°C using 1-hour exposure cycles followed by air blast quenching were used. Oxidation resistance was judged on the basis of weight change, as the first-order criterion and thermal fatigue crack resistance as a second-order criterion. The best coating-substrate system was judged to be the one with the greatest time to the maximum weight gain, t_1 . The following results were obtained:

1. Both bare alloys have t_1 values of less than 20 hours and thermal fatigue crack times of 40 hours.

2. Most of the coatings studied provided better oxidation protection for alloy VIA than for IN-100 both on a time to t_1 (weight change basis) and thermal fatigue cracking.

3. Based on time to t_1 (750 hr) the Pt-Al coating on VIA alloy was judged to be the most oxidation resistant system tested. This coating, however, does not provide superior thermal fatigue cracking protection. The second and third best systems were CoCrAlY coating on VIA and a cermet plus aluminide coating on VIA alloy. The times to maximum weight gain t_1 were 450 and 385 hours with thermal fatigue prevention to >600 hours and 600 hours, respectively.

4. The plasma spray applied FeCrAlY and NiCrAl coatings, as well as the slurry applied cermet coatings, exhibited poor adherence and failed by peeling from the surface, while the best coatings, as Pt-Al and CoCrAlY, failed by the slower, more common diffusion-spall process.

5. The weight change curves for the better systems are approximately parabolic in shape. That is, the weight of the specimen increases for a while - to time t_1 - and then decreases after reaching some maximum weight gain. The onset of the decrease in weight is associated with the start of the breakup of the coating layer, which leads to spalling. At a time $t_2 = \sim 2t_1$, the weight change curve crosses the zero axis and large weight losses occur. In general, thermal fatigue cracking occurred at times greater than t_1 .

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 10, 1973,
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TABLE I. - DESIGNATION, APPLICATION, AND GENERALIZED DESCRIPTION OF COATINGS

Coating	Method of application	Composition of coating materials used and heat treatment	General description of coating layer
MCrAl coatings			
FeCrAlY	Plasma sprayed	Fe-25Cr-13Al-0.8Y spray powder, oversprayed with Al then sintered 6 hr in Ar at 1100° C	Fe (solid solution) outer layer with β -NiAl, β -FeAl beneath; cracks and porous
NiCrAl (A)	Plasma sprayed	Proprietary composition, no added oxide	Coating porous (spongy); major surface phase, β -NiAl with minor phase γ -Ni ₂ Al
NiCrAl (B)	Plasma sprayed	Proprietary composition, Y ₂ O ₃ addition	Coating porous; major phases, β -NiAl and α -Al ₂ O ₃ ; Cr ₂ Si may be present
CoCrAlY	Physical vapor deposited	Co-22Cr-14Al-0.1Y, deposited layer - sintered 4 hr in H ₂ at 1080° C	Co (solid solution) with elongated β -CoAl grains dispersed
Cermets coatings			
Cermet (1)	Slurry dip	TiSi ₂ and TiN particles dispersed in NiCrSi alloy; sintered 1 hr in vacuum at 1150° C	Ni (solid solution) containing TiN and Ni ₁₆ Cr ₆ Si ₇ dispersed
Cermet (2)	Slurry dip	TiB ₂ and Al ₂ O ₃ particles dispersed in NiCr alloy; sintered 1 hr in vacuum at 1150° C	Ni (solid solution) containing Al ₂ O ₃ and TiB ₂ dispersed
Cermet (1A)/IN-100 ^a	Process for cermet (1) then Al pack at Lewis	Al pack was 2 wt. % Al, 2 wt. % NaCl, and 96 wt. % Al ₂ O ₃ heated for 15 hr at 1090° C in Ar; 15.3 mg/cm ² Al pickup	α -Al ₂ O ₃ dispersed in β -NiAl surface layer
Cermet (2A)/VIA ^a	Process for cermet (2), then Al pack at Lewis	Same processes for cermet (1A); 14.3 mg/cm ² Al pickup	β -NiAl surface layer formed
Cermet (1B)/VIA ^a	Process used for cermet (1) then an Al pack treatment at Lewis	Same process as for cermet (1A) coating; 13.0 mg/cm ² Al pickup	β -NiAl and α -Al ₂ O ₃ surface layer
Modified aluminide coatings			
Pt-Al	Electrodeposition and aluminiding	Electroplated Pt (0.00076 cm) and aluminided	Pt-Al rich outer layer over a CrNi alloy inner layer; layer sequence is PtAl ₂ , PtAl ₂ + β -NiAl, β -NiAl, then Cr, Ni, Ti, Al layer at metal surface
EAPA	Pack cementation aluminiding	Modified Al pack so that Al ₂ O ₃ as well as Al are deposited	NiAl containing about 15 vol. % of 2- μ m Al ₂ O ₃ particles
Aluminide	Pack cementation aluminiding	Proprietary Al pack for 6 hr at 980° C	β -NiAl and Ni ₂ Al ₃ layer formed with some cracks

^aApplied only to designated alloy. All other coatings applied to both VIA and IN-100 alloys.

TABLE II. - WEIGHT CHANGE PARAMETERS AND THERMAL FATIGUE DATA ARRANGED
IN THE ORDER OF INCREASING VALUES OF PARAMETER t_1

Coating designation	Substrate alloy	Time to maximum weight gain, t_1 , hr	Time till weight change curve crosses zero axis, t_2 , hr	Weight gain at t_1 , mg	Slope, mg/hr	Time to first thermal fatigue crack TTFC, hr	Nominal coating thickness before oxidation, cm
Bare	VIA	^a ND	ND	---	-1.1	40	----
	IN-100	↓	↓	---	-2.7	40	----
Cermet (1)	VIA	↓	↓	---	-7.5	>100	0.011
	IN-100	↓	↓	---	-9.2	>100	.013
Cermet (2)	VIA	↓	↓	---	-14	60	.008
	IN-100	↓	---	---	-25	60	.008
Cermet (1A)	IN-100	20	184	70	-.43	225	.018
Cermet (1B)	VIA	20	67	140	-2.9	>180	.015
Commercial aluminide	IN-100	20	220	25	-.12	200	.011
NiCrAl (A)	IN-100	40	68	130	-4.6	194	.023
Commercial aluminide	VIA	60	190	35	-.27	>300	.013
EAPA	VIA	60	205	11	-.08	>400	.005
FeCrAlY	IN-100	80	156	116	-1.5	>200	.019
FeCrAlY	VIA	100	180	116	-1.5	180	.018
NiCrAl (B)	VIA	100	325	340	-1.5	477	.023
NiCrAl (A)	VIA	120	230	180	-1.6	100	.015
EAPA	IN-100	140	270	17	-.13	120	.003
NiCrAl (B)	IN-100	150	^b 290	350	-2.5	240	.010
CoCrAlY	IN-100	160	245	27	-.32	>300	.020
Pt-Al	IN-100	380	725	44	-.13	^c 425 ^d 760	.008
Cermet (2A)	VIA	385	440	14	-.25	^d 600	.012
CoCrAlY	VIA	450	^b 1000	47	-.08	>600	.014
Pt-Al	VIA	750	^b 1400	48	-.07	^c 425 ^d 600	.008

^a t_1 and t_2 not determined (less than 20 hr).

^bExtrapolated value.

^cTE means crack observed on trailing edge of test bar.

^dLE means crack observed on leading (beveled) edge of test bar.

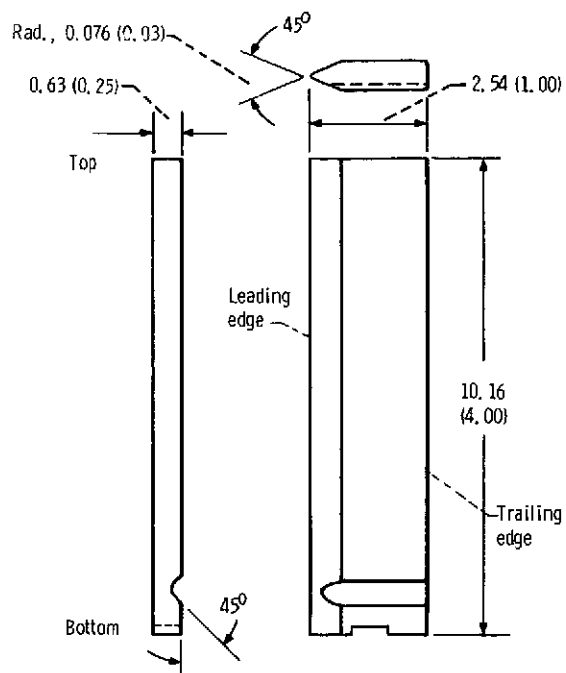


Figure 1. - Wedge bar test specimen. (Dimensions are in cm (in.).)

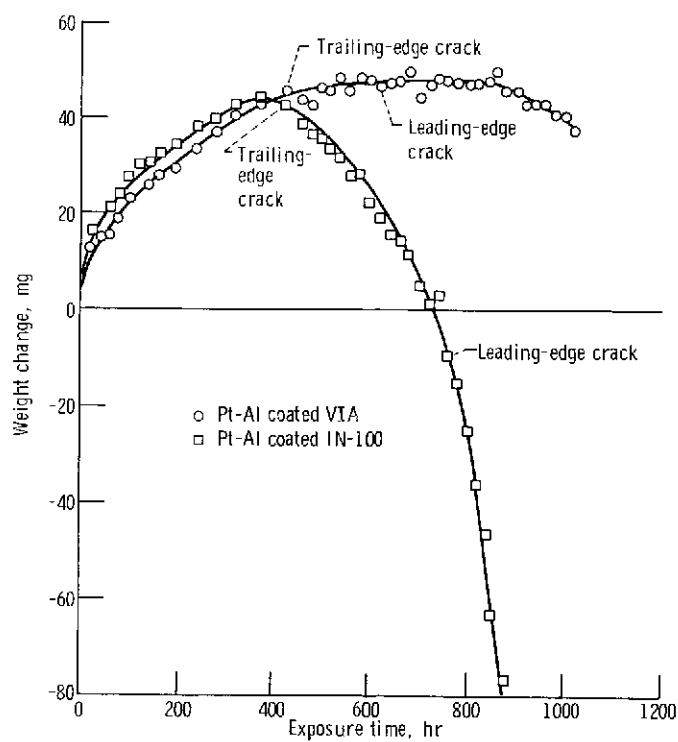


Figure 2. - Comparison of high-gas-velocity cyclic oxidation behavior of Pt-Al coated IN-100 and VIA alloys at 1093°C (2000°F).

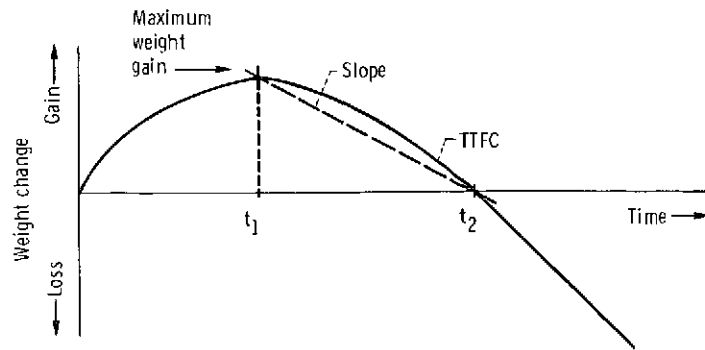


Figure 3. - Schematic diagram of weight change curve for high-gas-velocity cyclic testing of coated superalloys. Time to maximum weight gain, t_1 ; time at which weight curve crosses zero axis, t_2 ; slope is the slope of the straight line between t_1 and t_2 ; TTFC is the time at which first thermal fatigue crack is detected.

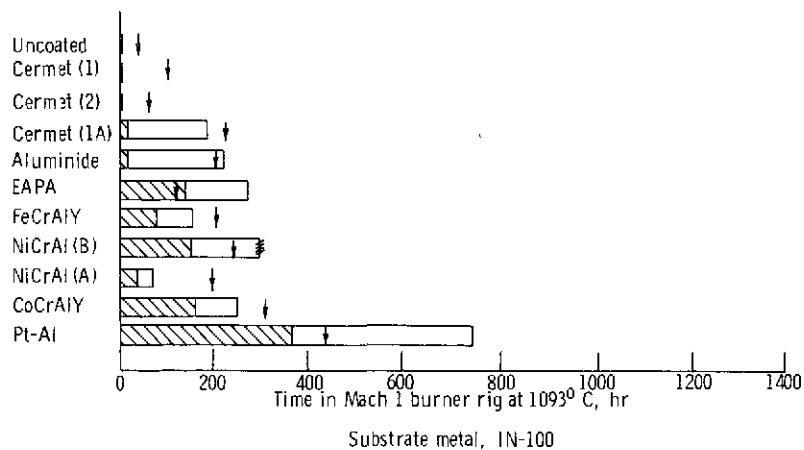
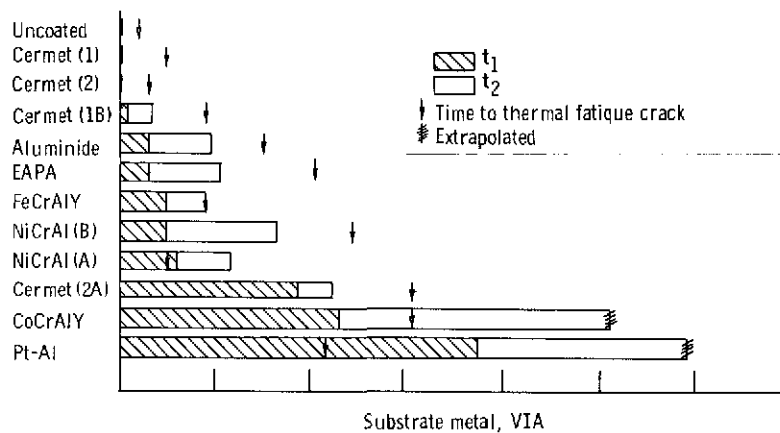


Figure 4. - Comparison of the oxidation parameters.

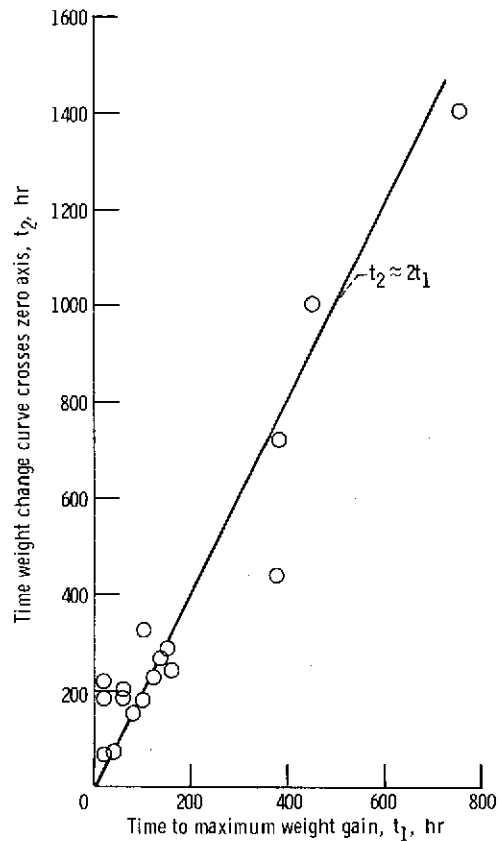


Figure 5. - Relationship between parameters t_1 and t_2 .

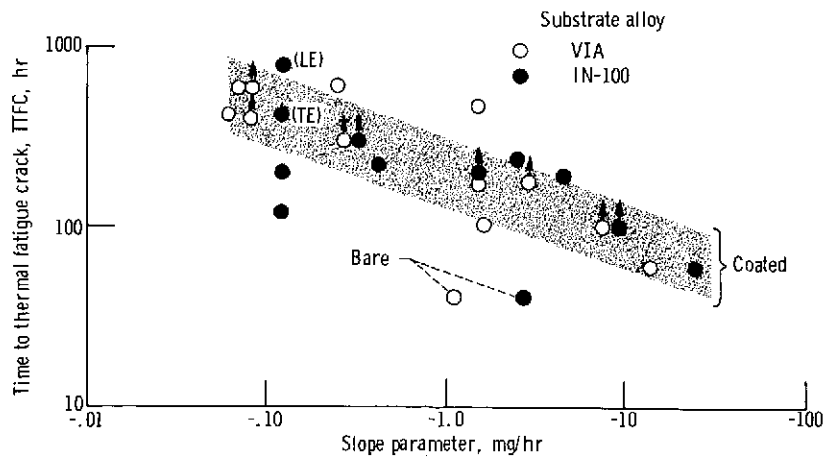
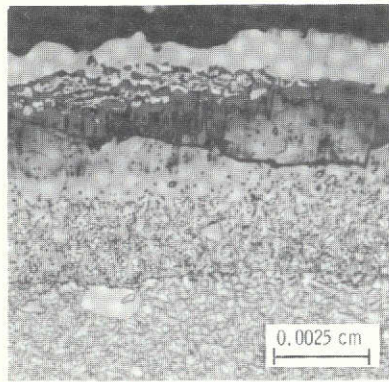


Figure 6. - Thermal fatigue crack time as a function of slope parameter of weight change curve for coated and bare IN-100 and VIA alloys cyclically oxidation tested at Mach 1 and 1093° C. (Note: Arrows indicate that TTFC is greater than time shown.)



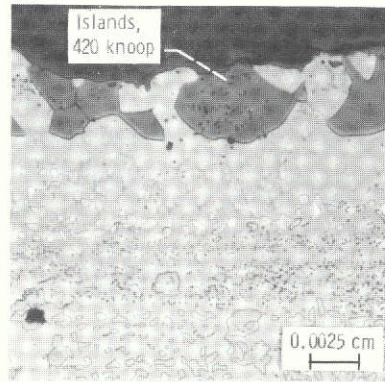
(a) As coated.

— PtAl₂ + βNiAl,
500 knoop

— β-NiAl,
520 knoop

— Cr, Al, Ti,
Ni alloy,
905 knoop

— Substrate,
560 knoop



(b) After 1020 hours of cyclic oxidation at 1093°C using 1-hour cycles.

— Al₂O₃

— PtAl_x +
Ni, Cr, Al
matrix

— 600
knoop

— Ni, Cr, Al
matrix

— Substrate,
460 knoop

Figure 7. - Photomicrographs of Pt-Al coating on VI A alloy. Knoop hardness numbers for 50-gram load.

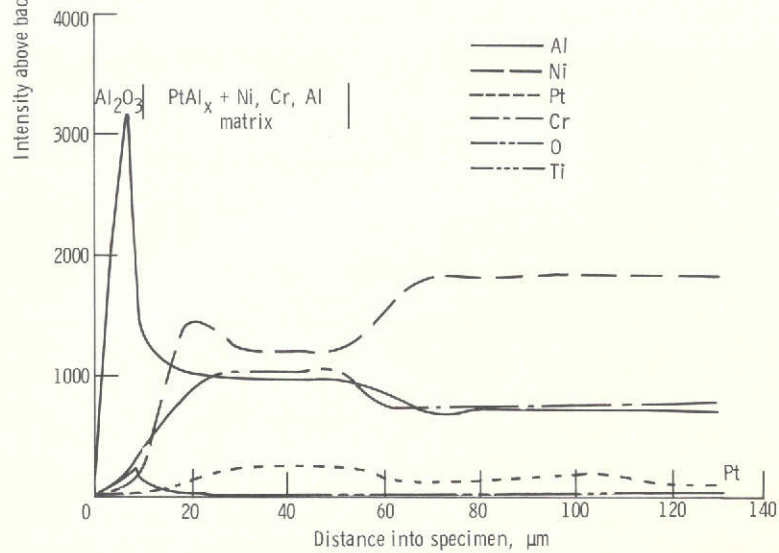
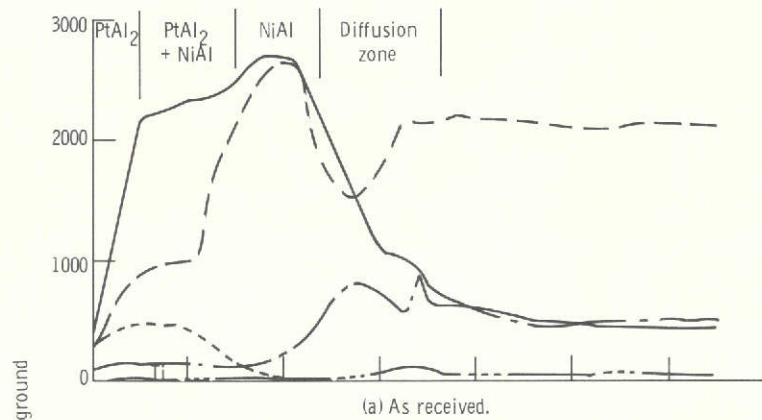


Figure 8. - Electron microprobe analysis of Pt-Al coated VIA alloy substrate. Integrated scan width, 160 micrometers.

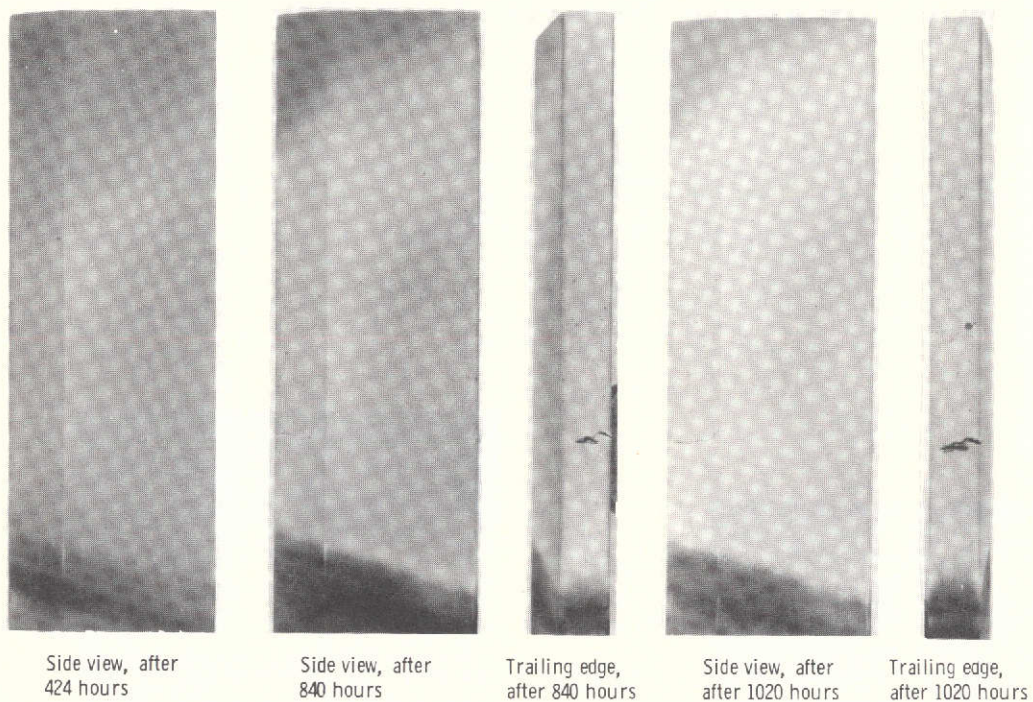
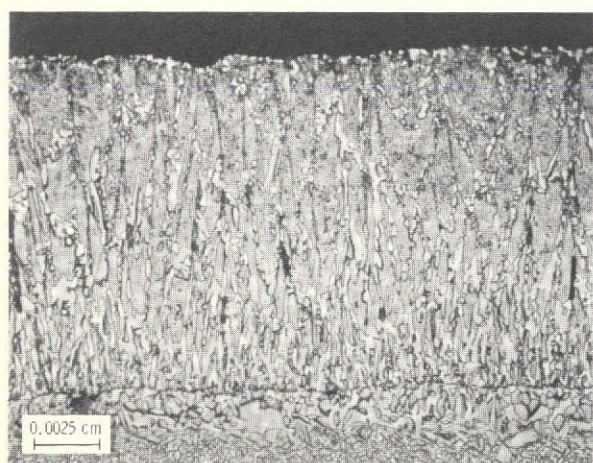
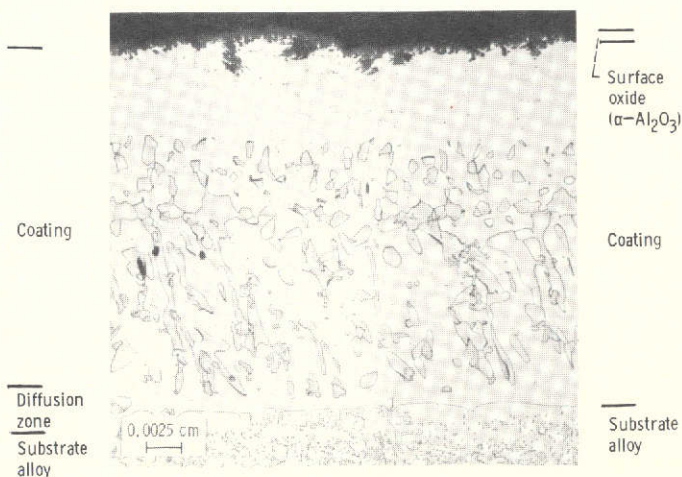


Figure 9. - Pt-Al coated VI A alloy after high-gas-velocity cyclic oxidation at 1093°C using 1-hour cycles.

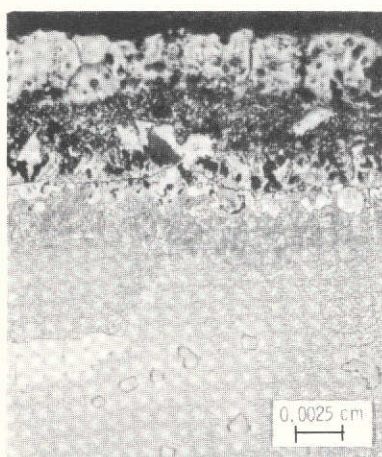


(a) As coated.



(b) After 600 hours cyclic oxidation at 1093°C using 1-hour cycles.

Figure 10. - Photomicrographs of CoCrAlY coating on alloy VI A.

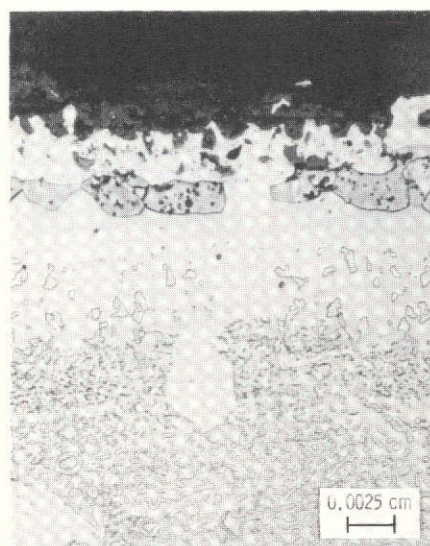


(a) As coated.

Aluminided
cermet
coating

Diffusion
zone

Substrate



(b) After 622 hours cyclic oxidation at 1093°C using 1-hour cycles.

Surface
oxide

Coating

Substrate

Figure 11. - Photomicrographs of cermet 2A coating on alloy VI A.



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—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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